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Study of Radiation Induced Electrical Degradation of Alumina in a Dynamic Pumping Condition in a Fission Reactor*

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The electrical conductivity of two ceramic insulators, alumina, and silicon nitride was measured in a fission reactor, JMTR under a dynamic vacuum condition. The instrumented irradiation rig was dynamically evacuated during fission reactor operation. The uppermost vacuum level attained was better than 10^{-2} Torr. In this experiment, we attempted to reveal effects of the gaseous environment on the measurement of electrical conductivity under ionizing irradiation. The results showed that the gaseous environment has a most hazardous effect in a certain gas pressure range.

In the second experiment, an electrical resistivity of polycrystal α-alumina was measured at about 680K in a dynamic pumping condition. In a good vacuum, we observed smaller RIC(radiation induced conductivity) than our previous results. We observed RIED(radiation induced electrical degradation)-like behavior. The results suggested that RIED would take off faster at a higher ionizing dose rate. In the meantime, the take-off occurred at about the same displacement damage of about 0.03-0.05dpa in the range of fast neutron flux of $3.4\text{-}15.1\times10^{13}$n/cm^{2}s.
Key words; electrical conductivity, α-alumina, silicon nitride, gaseous environment, in-reactor measurement in a dynamic vacuum.

1. Introduction

In-reactor measurements of electrical properties of ceramic insulators are currently attracting strong interests for fusion reactor developments. Some interesting results have been already reported.[1-3] However, the in-reactor measurement has many technical problems which sometimes jeopardize obtained results. Especially, hampering effects of irradiation environment is worried most seriously. We have been studying the dynamic effects of irradiation on the electrical conductivity of ceramic insulators, using a fission reactor, JMTR(Japan Materials Testing Reactor) in Oarai Research Establishment of Japan Atomic Energy Research Institute.

Up to now, we developed small subcapsules in which specimens were sealed in a dead vacuum. Using these subcapsules, we have measured the electrical conductivity in-situ several times in a fission reactor.[4-7] However, this experimental procedure evokes the apprehension that the deterioration of the vacuum in the subcapsule would affect the obtained results.[8] The effects of residual gas on the electrical conductivity have been well recognized[8], and their effect on the dielectrical property was demonstrated in a pulse type fission reactor with relatively large ionization dose rates[9].

Thus, we developed techniques for the in-situ measurement of electrical conductivity of ceramic insulators in a dynamic vacuum condition in a fission reactor. We established a tachnique for evacuating a whole irradiation rig in a reactor core in our first experiment. Using this technique, we studied the effects of gas and residual-gas environments on the measurements of electrical conductivity.

Our first experiment revealed that it is essential for successful in-situ electrical measurements that we have both a vacuum environment better than 10^{-2} Torr and good thermal contact between a specimen and a reactor coolant. These two conditions are not easily compatible with each other in a reactor core.

Here, we developed a subcapsule and we evacuated it in a reactor core in our second experiment to realize the two conditions mentioned above., instead of evacuating a whole irradiation rig in the previous experiment. In this well-controlled environment, we measured electrical conductivity of α-alumina and studied the degradation behavior of its electrically insulating ability along the irradiation.

In this paper, we will describe details of the developed procedure for irradiation in a dynamic vacuum condition in a reactor and will show the obtained results of the effects of gaseous environments on measurement. Also, the paper will report results in the second experiment and will compare them with those obtained previously in the same reactor.

2.Experimental Method

We irradiated sintered ceramic materials of 99.5% alumina, A-479SS, and silicon nitride, SN-220. Both were made by Kyocera Co. Ltd. Japan, in JMTR in a developed irradiation rig whose structure is shown in Fig. 1 schematically. The rig could be evacuated to a vaccum. The neutron fluxes were 7.0×10^{13} and 2.0×10^{14}n/cm^{2}s for the fast(E>1MeV) and the thermal (E<0.6826eV) neutrons, respectively. The γ-ray dose rate was measured by the calorimetry to be about 4×10^3 Gy/s at the reactor-full power of 50MWth.

In the JMTR irradiation, we need to connect the irradiation rig and pumping system with about a 20m long tube for the evacuation. The tube diameter is limited to be 5mm in its outer diameter and 4mm in its inner diameter. Also, we can not measure the gas pressure directly in the

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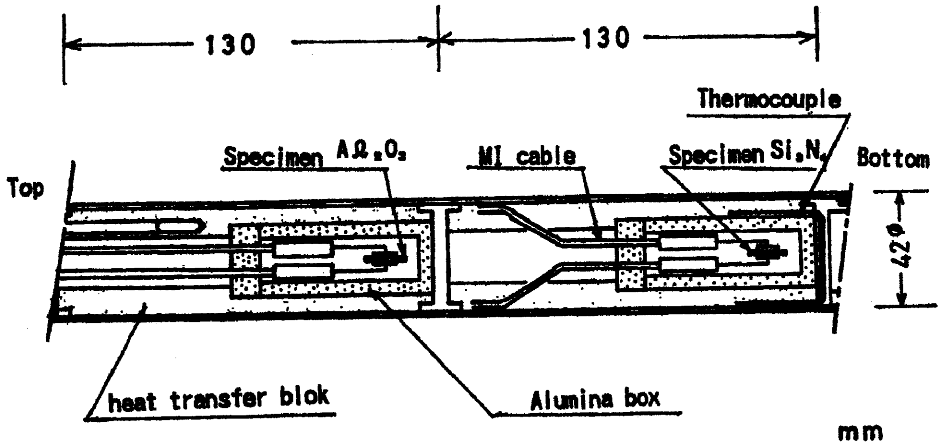


Figure1 Structure of the irradiation rig for measurement of electrical conductivity of ceramic materials

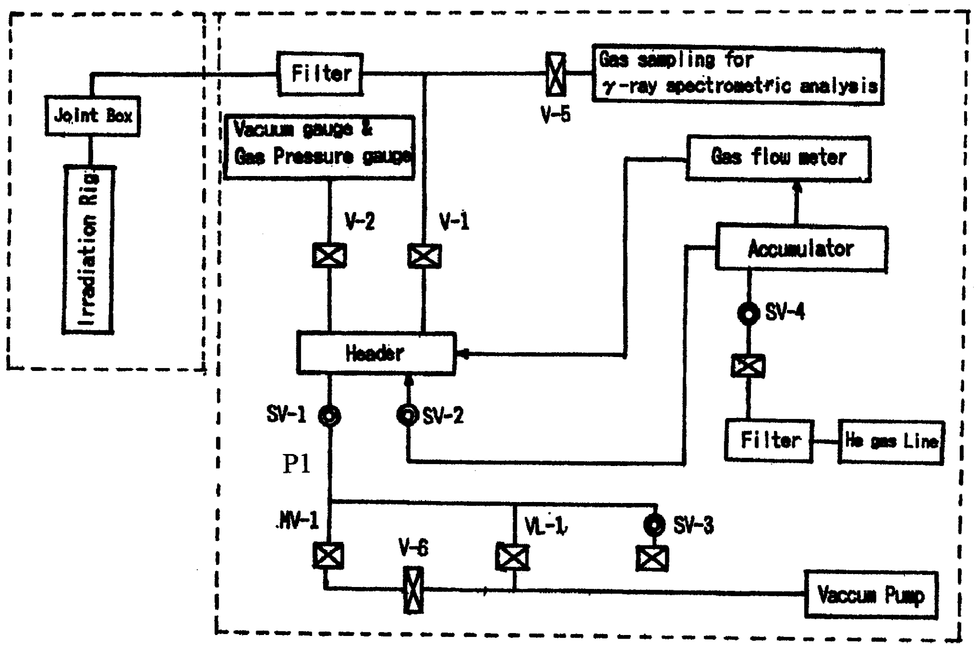


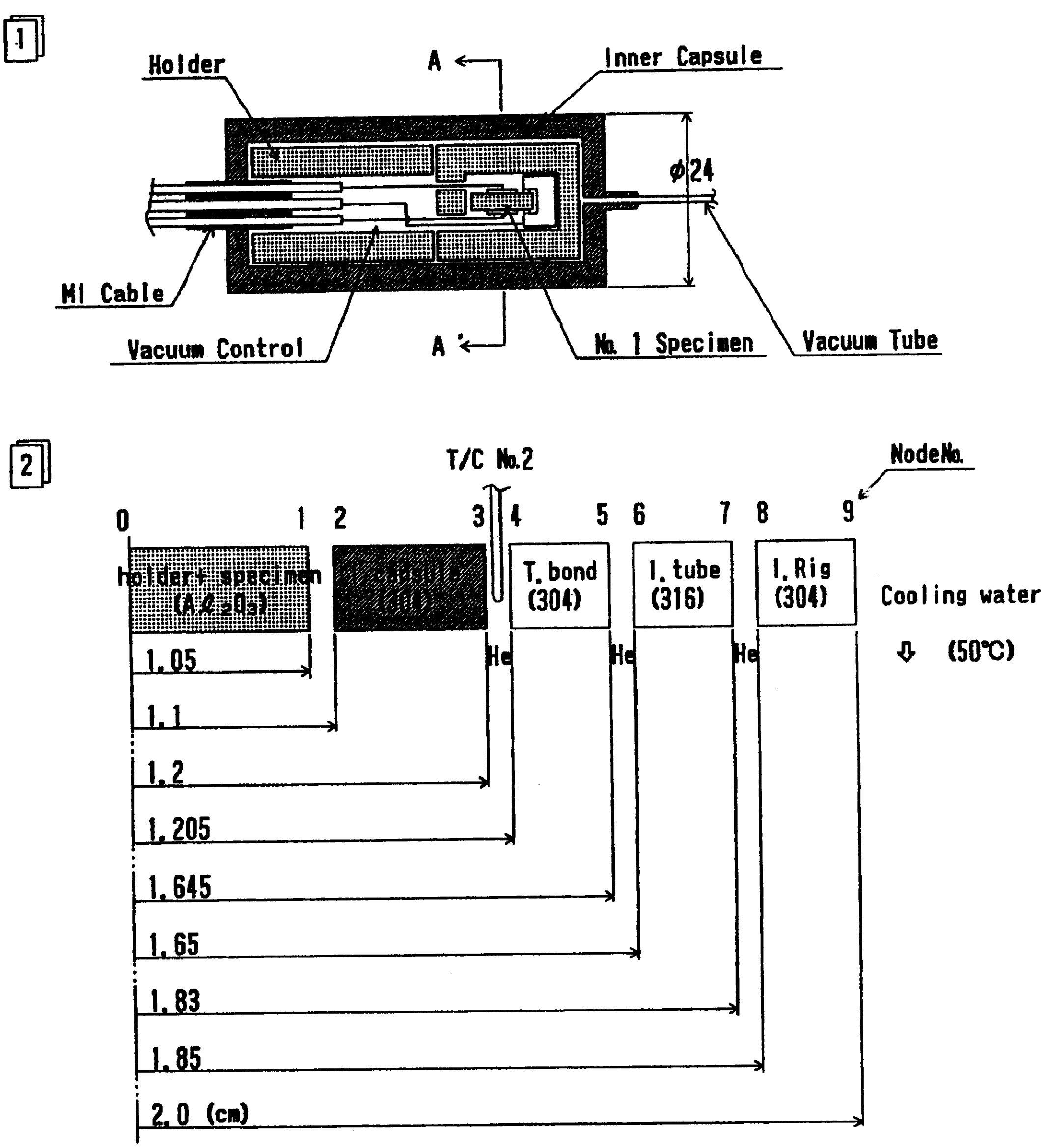
Figure2 Evacuation system for in-reactor irradiation rig.

irradiation rig inserted in the reactor core. So, we made the identical irradiation rig for the mock-up test in the cold(out of reactor). The rig was made of SUS 316 stainless steel tube whose inner diameter and length were 38mm and 1m, respectively. The evacuation test was carried out in the cold(out of the reactor core) and we measured actual gas pressures at the top(P2) and the bottom(P3) of the irradiation rig which contains actual inner structures and containers in it, as well as at the point P1 in Fig. 2.

The rig was evacuated in two ways. First, we evacuated the rig without any gas flow. Second, we evacuated the rig with flowing helium gas from the leak valve. The reason why we carried out the second type evacuation will be described in the next section. The actual structure of the evacuation line is shown in Fig. 2 for the in-reactor irradiation experiment. Before the reactor went operation, the irradiation rig was filled with 1.3atm helium gas. Then the reactor power was raised up to its full power of 50MWth, during which the helium gas pressure was kept at 1.3atm. The helium gas flow rate was kept at about 20cc/min. After the reactor operation was in the steady state of its full power for 18h, the valve SV-1 was open and the evacuation started. In this stage the helium gas flow rate was kept at the initial value of about 20cc/min. Then, the helium gas flow rate was decreased step by step to the final flow rate of less than 1cc/min.

The gas pressure was measured at the header in Fig. 2. When the gas pressure range was higher than several Torr, the absolute gas pressure gauge was used, and when the vacuum range was better than a few Torr, the Pirani-gauge was used for measuring the gas pressure. The structure of the present irradiation rig around the specimens is shown in Fig. 1. The specimen was accommodated in the small space made of alumina(Kyocera A-479), and was suspended in the empty space with two electrical platinum lead. It was designed that the walls facing the specimen should be composed of the same material-kind of the specimen. So, the temperature was measured only at the outer wall of the alumina-made small container as shown in Fig. 1. The specimen temperature was estimated from the thermo-couple signal by the calculation, using a one-dimensional heat flow model and taking into consideration the temperature drop across the gas gap between the specimen and the outer wall.

The electrical conductivity was measured by applying DC voltage up to 300 V and by measuring the electrical current by the picoammeter. Here, we did not adopt the three electrode configuration, namely, we did not attach the guard electrode. In the previous experiment[4], we judged that the surface current could be very small compared with the bulk current evoked by the radiation induced conductivity in the pre-evacuated condition.[7] However, it must be admitted that surface currents are sensitive to environments, namely, to the vacuum condition in the present experiment. The electrical conductivity of the silicon nitride was monitored



3

(℃)

Node No.	0	1	2	3	4	5	6	7	8	9
Calculated Temp.	411	409	342	340	318	293	243	226	79	59
Experimental Temp.	—			280 (T/C No. 2)		240				

Figure3 Cross-sectional view of developed subcapsule. Specimen is in a sub-room made of α-alumina and results of temperature estimation by GENGTC-code and their comparison with measured ones.

by the impedance analyzer of HP 4194A, at 100Hz. The electrical conductivity was measured as a function of the reactor power in the 1.3atm helium gas environment. Also, it was measured as a function of the helium gas pressure at the reactor full power.

Here, it should be noted that the specimen temperature changed by the change of the helium gas pressure, due to the alteration of thermal conductivity of the gas-filled gap. At the 1.3atm helium gas pressure, the specimen temperature was estimated to be about 670K. When the gas was evacuated to be about 0.5Torr, the specimen temperature was estimated to be about 880-900K. The temperature was estimated to be higher than 1300K when the gas was evacuated to less than 0.1Torr.

As shown below, the major results of the present experiment were obtained in the gas pressure range higher than 0.5Torr. The previous experiment showed that the electrical conductivity measured in the reactor was temperature-independent in the range up to 870K. So, the major part of the present results was not affected by the temperature change. However, at the gas pressure range lower than 0.1Torr, the results were affected significantly by the temperature rise. After a few days irradiation in a vacuum better than 0.1Torr at the reactor full power, a significant electrical current leak occurred, and the experiment could not be continued further. This failure was probably due to the evaporation and the deposition on the electrical insulators of the metallic atoms at elevated temperatures. We used a brazing agent to make good bonding between the platinum electrodes and the specimen. The brazing agent contained silver, which had high vapor pressure at elevated temperatures.

In the second experiment, we developed a subcapsule. In a SUS316-made subcapsule, a specimen was encased in a small sub-room composed of all alumina-made parts. The specimen was a polycrystal α-alumina, Kyocera A479ss of 99.5% purity, 8mmφ and 0.15mm thick. A three-electrodes configuration was adopted and a guard-ring electrode contacted to a SUS316-made heat sink block, which had good thermal contact to coolant water of about 330K through the wall of subcapsule. The platinum electrodes were blazed to the specimen. The typical resistance of the electrodes including 40m MI-cables was about 30-50Ω. The resistance between two electrodes and a guard ring electrode were measured to be better than 10^{12} Ω at room temperature before irradiation. The diameter of measuring electrode is 2mm.

This subcapsule was evacuated through 3mmφ and 25m long tube during irradiation. The total surface-area inside of the subcapsule was about $100cm^2$, being less than 1/20 of that of our previous experiment, where a irradiation capsule of 38φx750mm long was evacuated. A mock-up test out of the reactor core showed that vacuum of about 10^{-5} Torr could be attained in the present system, at 773K. he subcapsule was accommodated in a temperature controlled irradiation rig[10] and was inserted into the reactor core. The reactor used was JMTR. Evacuation of the subcapsule started one week before the reactor start-up and continued to the end of the experiment.

he estimated γ-heating rate is about 4.5W/g for alumina, namely $4.5x10^3$Gy/s. Neutron fluxes were $1.2x10^{14}$ and $2.0x10^{14}$ n/cm^2s for fast(E>1MeV) and thermal(E<0.683eV) neutrons, respectively. The displacement damage rate is estimated to be $1x10^{-7}$ dpa/s.[11] Irradiation temperature was monitored at the outer surface of the subcapsule as shown in Fig.3. he temperature of the irradiated specimen was estimated from a calculation using the GENGTC code.[11] Estimated temperatures at some points in the irradiation rig agreed well with measured ones as shown in Fig.3 which will vindicate validity of the estimation. The estimated temperature at the specimen was 680K.

A DC voltage of 100V was continuously applied to the specimen and the electric current through the specimen was measured by a picoammeter. In the course of the experiment for 71 days with 29rfpd(reactor full power days) and 3 intermissions of 42 days in total, the measurements were stable and reproducible.

3. Results and Discussions

In the cold(out of reactor) test, we measured the gas pressure at three points. Fig. 4 shows the measured gas pressures of P2 and P3 as a function of the gas pressure at the point P1. When the leak valve of L1 was closed and there was no helium flow, the vacuum in the irradiation rig reaches the value at better than 10^{-2} Torr. Thus, it was proven that we could evacuate the irradiation rig to a relatively good vacuum even with the long and fine evacuation line of 20m long and 4mmφ assuming that the rig was well outgassed and the outgassing rate of the rig was small. Also, the gas pressures at the top and bottom of the irradiation rig were not significantly different. So, the gas pressure in the irradiation rig is relatively uniform. However, the pressure difference between P1 and P2&P3 is

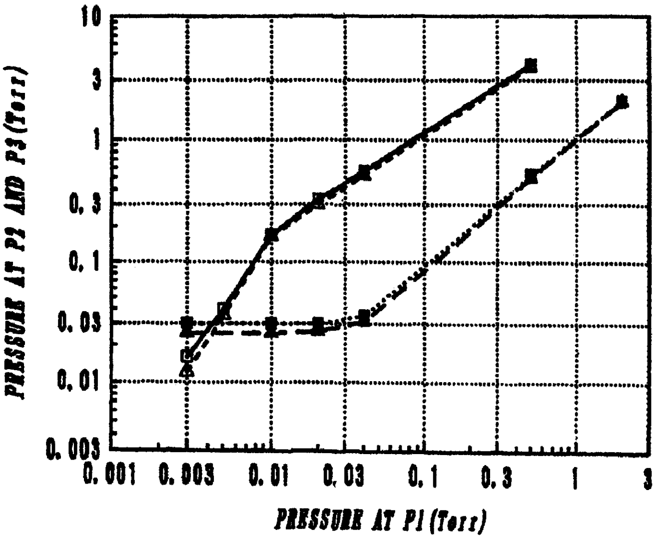


Figure4 Gas pressure relationship between different positions of the irradiation rig as a function of gas pressure at P1. Triangle; P2, square; P3, open; without gas flow, closed; with gas flow.

quite significant. The pressure at P2&P3, namely the pressure in the irradiation rig is about one order of magnitude higher than the point P1, although the difference became smaller as the pumping continued. After the 70 h pumping, the pressure at P2&P3 became only about 3 times higher than that at P1.

When the leak valve was open and some amount of the helium gas was flowing, the pressure difference between P1 and P2&P3 became quite small , when the pressure at P1 is higher than $4x10^{-2}$ Torr as shown in Fig.4. In this pumping condition, the pressure at P1 is a good indicator of the

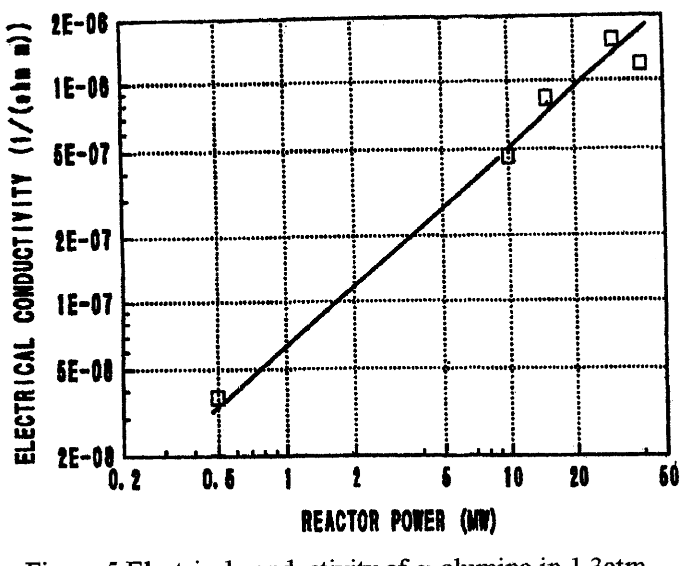


Figure 5 Electrical conductivity of α-alumina in 1.3atm helium gas as a function of reactor power.

pressure in the irradiation rig. In the actual pumping of the irradiation rig in the reactor, some amount of the helium gas flows through the header, and it is considered that the pressure measured at the header is a good indicator of the gas pressure in the irradiation rig in the reactor.

Figure 5 shows the electrical conductivity of the alumina specimen measured in the 1.3atm helium environment at the start up of the reactor. The measured conductivity changed with the reactor power nearly linearly. More precisely, the relationship is a slightly sublinear. This increase of conductivity with the reactor power is thought to be due to the radiation induced conductiviy[2]. Compared with the previous results[4-5] using same grade of Al_2O_3 specimens, the measured conductivity was a little larger especially at smaller reactor power. This difference may be due to the

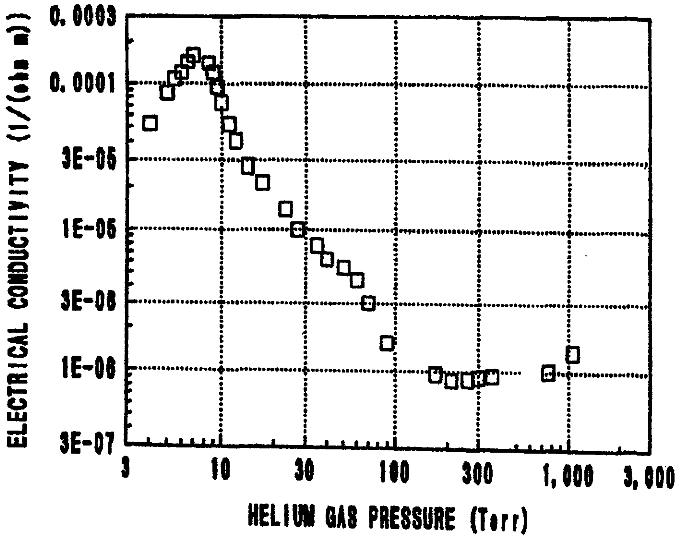


Figure 6 Measured electrical conductivity of α-alumina at the reactor-full-power as a function of the helium gas pressure.

1.3atm helium gas environment. The sublinear relationship between the conductivity and the reactor power, in contrast with the previously obtained slightly superlinear relationship, also suggested the helium-gas environmental effect.

When the pumping was started and the helium gas flow rate was decreased, the electrical conductivity changed. Fig.6 shows the conductivity change as a function of the helium gas pressure at P1. As described above, the gas pressure at P1 is thought to represent the gas pressure in the irradiation rig well as far as the helium gas flows. Initially, the measured electrical conductivity decreased with decrease of the helium gas pressure. The conductivity attained its minimum at the helium gas pressure of about 230Torr and then increased rapidly. This behavior was also observed for the AC conductivity of the silicon nitride at 100Hz. The minimum conductivity of Al_2O_3 at about 230Torr was 9.6×10^{-7} $(\Omega m)^{-1}$. This value fits well with the results measured in the previous experiment using the evacuated subcapsule[5,6].

The electrical conductivity of alumina increased to a value of $1.6\times10^{-4}(\Omega m)^{-1}$ at the helium gas pressure of about 7Torr and then decreased with decrease of the gas pressure. The DC conductivity measurement of the alumina was carried out to the point of the helium gas pressure of about 3Torr, where the measured conductivity had decreases to the value of 5×10^{-5} $(\Omega m)^{-1}$. At this point, the measurement became quite unstable, and then the measurements could not be carried out any more. The current leads were actually grounded.

In the meantime, the AC conductivity measurement on the silicon nitride was continued. The AC conductivity increased further to a value of more than 3×10^{-1} $(\Omega m)^{-1}$ at the helium gas pressure of about 2.5×10^{-1}Torr. Then the AC conductivity of the silicon nitride deceased with decrease of the gas pressure to a value smaller than 3×10^{-4} $(\Omega m)^{-1}$. At this point, the current leads were grounded, and measurements could not be continued.

The electrical conductivity of the alumina and the silicon nitride did not change by the conditions of the surrounding gas environment significantly. So, the observed change of the electrical conductivity was due to the parasitic effects of the gas environment. The present experiment indicated that a gas environment affects the electrical conductivity measurement significantly at a certain pressure range. In the DC current measurement, the effect of the helium gas environment is the most significant in the pressure range near 5Torr. In the meantime, the helium gas pressure near 0.2Torr affected the electrical measurements of AC conductivity at 100Hz most significantly.

In the gas pressure range higher than the described critical pressure range, the measured electrical conductivity was less sensitive to the gas pressure but still it had distinct gas-pressure-dependence. In this experiment, it is thought that the helium gas pressure of about 200-250Torr is the most desirable environment, where we obtained the lowest electrical conductivity in both the DC and the AC measurements.

In the pressure range lower than the critical gas pressure range, we also have the gas pressure range where the electrical measurement is not affected by the environment. Unfortunately, the present experiment does not show this region distinctly, although the results suggested that a gas pressure less than 10^{-1}Torr does not affect the present measurement significantly in the case of the DC measurement. For the AC measurement, a vacuum better than 10^{-2}Torr is needed.

There is a famous law called Pashen's law on the dielectric breakdown potential of gases in the absence of ionizing radiation.[12] The law describes the electrical breakdown voltage as a function of P*D. Here, P is the gas pressure and D is a distance between two electrodes. In the case of the air, the minimum voltage needed for the electrical breakdown is reported to be 300V when P*D is 0.3-0.7Torrcm. In general, Pashen's law indicates that the air is the most electrically-conductive at the pressure of about 5Torr.In the absence of ionizing radiation, the electrical breakdown of a gas will take place through complicated processes such as an acceleration of gas ions and electrons, subsequent ionization and neutralization of gas atoms, formation of secondary electrons on the electrodes by the bombardment of energetic ions and electrons , etc..

Under the strong ionizing irradiation, there would already exist many ions and electrons, that would make the process of electrical breakdown of a gas different from that in the absence of ionizing dose. One distinct difference we could point out is that a high voltage is needed in the absence of ionizing radiation, while even the application of 0.5V caused the electrical breakdown in the present ionizing radiation environment. The effect of the frequency of applied voltage was also studied on the electrical breakdown of gas and vacuum environments in the absence of ionizing radiation.[12] It was reported[12] that the 50 Hz AC voltage has a higher breakdown voltage than the DC voltage. However, in general, it was recognized that the frequency of applied voltage affects slightly on the breakdown voltage in the frequency range of 60Hz-45MHz[12]. These results would imply that the observed difference between the DC and the AC measurement in the present experiment should be examined further more.

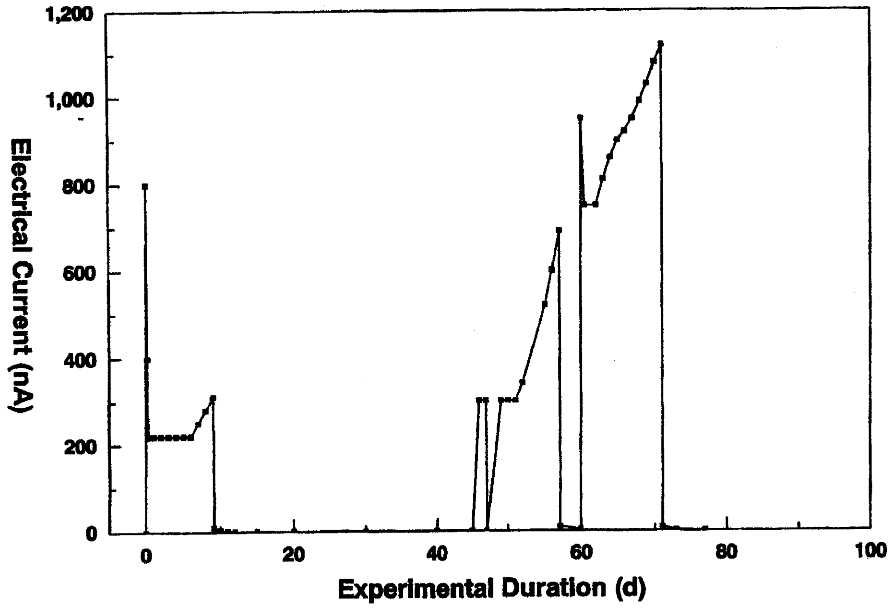


Figure 8 History of measured electrical current in the course of experiment.

The results obtained in the higher helium gas pressure range agree well with the previously obtained results[4]. This suggested that the previous results were not have affected strongly by the residual gas in the subcapsule. In retrospect, the results obtained in our first experiment, where we measured the resistivity mainly by the AC method, might have been affected by the residual gas in the subcapsule.

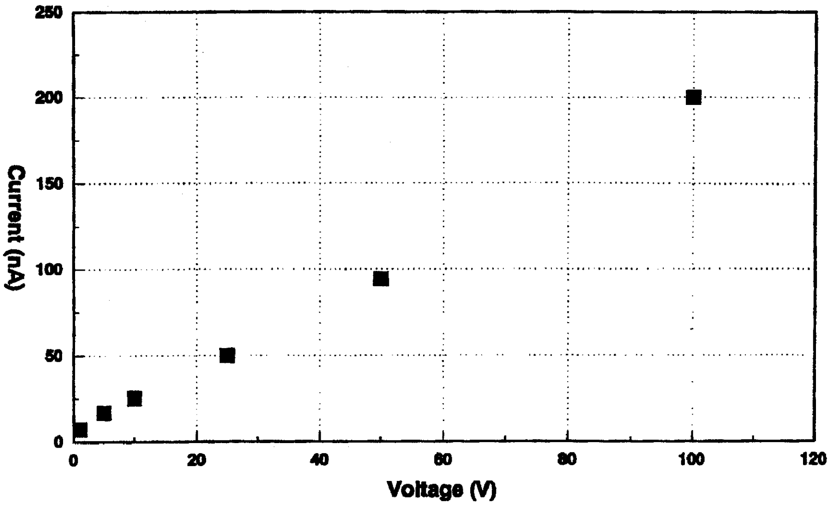


Figure7 Measured electrical current as a function of applied voltage at 50MW.

In the second experiment, an electric current of 23pA was measured to go through the specimen with a 100V DC voltage at room temperature before start of the reactor irradiation,. At the start-up of the reactor, the electrical current increased with the reactor power. Just after the reactor power reached its maximum power of 50MW, the current was measured to be 500-800nA. The observed current was nearly ohmic but showed some deviation as shown in Fig.7.

The measured current decreased rapidly to the value of 200-220nA within several hours after the reactor power reached 50MW. Then the measured current kept nearly constant at 200-220nA for 5 days. The observed electrical current of 200-220nA will correspond to a conductivity of $1 \times 10^{-7} Sm^{-1}$. Compared with our previous results, this value is a little smaller. We obtained a value of $1 \times 10^{-6} Sm^{-1}$ for the same specimen of 0.5mm thick at 770-800K in a γ-ray dose rate of 5.3×10^{3}Gy/s. This experiment was carried out in a so-called dead-vacuum condition[5]. Also, in a 300Torr helium environment, we measured a conductivity of $8\text{-}9 \times 10^{-7} Sm^{-1}$ at a γ-ray dose rate of 4×10^{3}Gy/s.[4] From these results, we expected the conductivity of $5\text{-}10 \times 10^{-7} Sm^{-1}$.

The observed smaller electrical conductivity rather agree with reported data using ions[13] and may imply that the previous measurements would be affected by their environments. The observed decrease of electrical current at the initial stage of the present irradiadeteriorate tion may also support this. The initial increase of reactor power will increase the degassing rate and the vacuum in the subcapsule, but the degassing rate will decrease soon. Subsequently, the degree of vacuum would be improved soon.

After 6-days irradiation, the current began to increase. The current reached to the value of 300-310nA just before the first reactor-stop took place after 9-days irradiation. During the succeeding 36-days intermission period, the current decreased from 10 to 1nA. This decrease of current

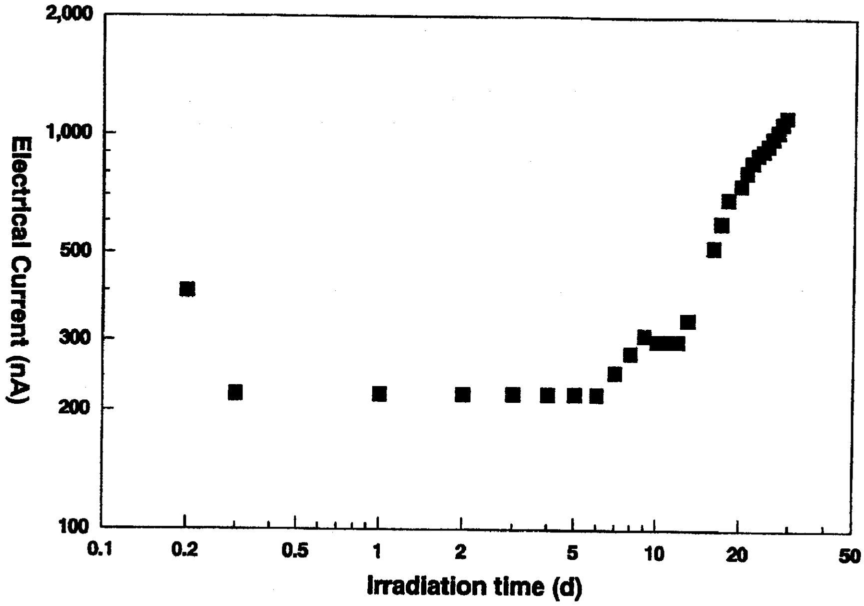


Figure 9 Log-log plot of electrical current vs. irradiation duration.

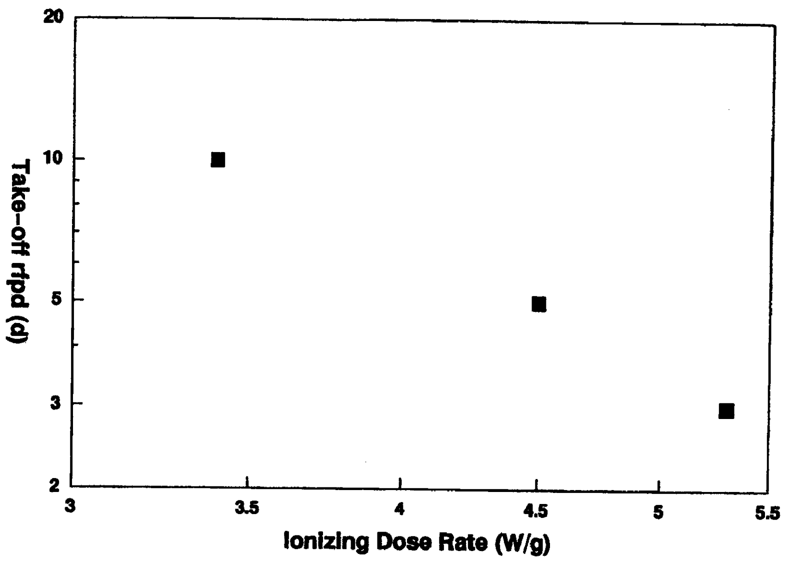


Figure 10 Take-off duration of observed RIED as a function of the ionizing dose rate.

will be due to the decrease of the dose rate of residual γ-ray in a reactor core.

At the next reactor-start-up, the current again increased to 280-300nA, a little smaller than the previous value. Then, the reactor stopped after a one-day-long operation. At the reactor-stop, the measured current again decreased to 1nA. After 2-days intermission, the reactor again started, and the observed current increased to 280-300nA. The current kept to increase for another 8-days irradiation, to a value of 700-710nA. At the final start-up of reactor, the current attained the previous value of 680-700nA.

Then again, the current continued to increase up to the end of the irradiation. The detailed current change described above is depicted in Fig.8. After the final reactor-shutdown, the observed current decreased from 10nA to 1nA for 6days, which is similar to the behavior observed in the first intermission. The measured electrical current was shown in Fig.9 as a function of reactor full power days. The behavior of current increase is similar to the previously observed ones, which were thought to be described as RIED, radiation induced electrical degradation[14]. However, we must admit that the electrical conductivity decreased to a low level after the reactor stopped as shown in Fig4. This behavior is different from RIED observed in other irradiation fields[13,14], where the increase of electrical conductivity due to RIED sustained even after an irradiation has ceased. In these series of our experiment, we observed only moderate degradation of electrical insulating ability as shown in this experiment. So, we might have observed only an initial stage of RIED, where permanent degradation might not be definite.[15]

Fig. 10 shows the take-off time of RIED as a function of the γ-ray dose rate in series of experiments[4,5]. Four in-situ experiments have been carried out in three different irradiation positions in the reactor up to now. The take-off time has a -2.8(smaller than -1) power dependence on a dose rate. This means that a higher dose rate makes the take-off faster even in terms of a total dose.[16] In the meantime, the take-off took place at the total displacement damage of about 0.035-0.05dpa in these three experiments, where the fast neutron flux ranged in 3.4-15.1x10^{13}n/cm^2s. Here, it should be noted that, as described above, the previous results would have be affected by environments effects. We would have observed higher RIC, which might have delayed our observation of RIED.

4. Conclusion

We developed the irradiation rig for the in-situ measurement of the electrical conductivity under the dynamic vacuum condition. We measured the electrical conductivity of the alumina and the silicon nitride in variety of the helium gas pressure. In the gas pressure range higher than a few 100 Torr, the helium gas pressure had only a slight effect on the measurement. The gas pressure of about 200-250Torr is thought to be the best pressure for the measurement in the pressure range of 10-2 to 1000Torr.

The helium gas pressure of about 5Torr is thought to be the most harmful to the DC measurement, while, that of 0.1-0.3Torr is the most hazardous to the 100Hz AC measurement. Gas pressures one order of magnitude smaller than these values may also be good for a reliable measurement of the electrical conductivity in a fission reactor environment.

We measured electrical conductivity of α-alumina in a good vacuum condition in-situ in a fission reactor, JMTR. We observed radiation induced conductivity, RIC, whose magnitude is smaller than values obtained in our previous experiments. This may imply that environment effects would be more serious than previously assumed.

High pressure helium may also have some unfavorable effects. We also observed, RIED(radiation induced electrical degradation)-like behavior in the course of irradiation. Comparing the present results with previous results, it is suggested that RIED would take off faster at a higher dose rate.

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